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Preparation of magnesium hydroxide nanoflowers from boron mud via anti-drop precipitation method

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ABSTRACT

Using boron mud as the starting material, the flower-like magnesium hydroxide (MH) has been successfully prepared via anti-drop precipitation method. The effect of $NH_3{\cdot}H_2O$ concentration, aging time, and surfactant on the morphology of MH was investigated. The optimum precipitation conditions are dropping $MgSO_4$ solution in 5% $NH_3{\cdot}H_2O$ solution, with 3% polyethylene glycol as surfactant, aging for 30 min. XRD, SEM, FI-IR, and TG/DTA have been employed to characterize the as-prepared samples. XRD reveals that MH with high purity has the brucite structure. SEM images show that the flower-like MH exists in the form of mono-disperse well uniform spherical aggregation with diameter of 3–5 μm. TG/DTA shows a total percentage of weight loss 33.6% with a well-defined endothermic peak near 381.3 °C corresponding to the decomposition of MH. Furthermore, it reports that the extremely fast primary nucleation is of significance for crystal growth of MH.

1. Introduction

Magnesium hydroxide (MH) is a popular inorganic compound because of its wide range of applications. It plays an important role in many fields, such as flame retardant in polymers [1–3], special ceramics [4,5], fillers in bleaching agent [6,7], wastewater treatment [8–11], and so on. Furthermore, it is a raw material for preparation of magnesium oxide [12,13].

Many methods can be used for synthesis of MH, including hydrothermal method [14,15], solvothermal method [16], sol–gel technique [17], cathodic electrochemical process [18], microwave and ultrasonic assisted techniques [19–21], and precipitation [22–26]. Comparing with these methods, the precipitation method is the most inexpensive and green method for synthesis of MH.

MH nanoparticles have been synthesized by the above-mentioned methods using different magnesium sources. The chemicals magnesium nitrate ($Mg(NO_3)_2$), magnesium chloride hexahydrate ($MgCl_2{\cdot}6H_2O$), and magnesium acetate ($Mg(CH_3OO)_2$) are the commonly used raw materials [18,20,23]. However, the minerals dolomite, brucite, and magnesite with high content of magnesia as magnesium sources have been reported in the literatures [13,14,27]. Boron mud with about 40% magnesia is a potential magnesium source for synthesis of MH.

Shape and crystal size of MH affect its applications, for example the flower-like MH nanoparticles which have higher decomposition temperature are used as a flame retardant in the composite materials [2,15]. Generally, the shape and crystal size are strongly depended on the preparation process and the precursors [22,23,26,28–30]. Meanwhile, surfactants have been widely used to control the shape and crystal size of MH nanoparticles, and polyethylene glycol used as surfactant was reported in the literatures [23,28].

In this work, the flower-like MH has been prepared via anti-drop precipitation method using the boron mud powder as the starting material and ammonium hydroxide as precipitant with polyethylene glycol as surfactant. Furthermore, the formation mechanism of the flower-like MH via anti-drop precipitation method has been proposed.

2. Experimental

2.1. Materials

The boron mud powder was used as the starting material, which is solid waste from a borax factory in Liaoning province, China. The major chemical compositions of the boron mud powder are MgO 43.36%, SiO_2 25.99%, and Fe_2O_3 5.55%. Chemical analysis and XRD analysis indicate that the principal minerals of the boron mud powder are 54.0% olivine, 29.5% magnesite, and 5.6% phlogopite [31]. The sulfuric acid used was of analytical reagent

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grade with a purity of 98%. The $NH_3{\cdot}H_2O$ was of analytical reagent grade with a purity of 25–28%. Polyethylene glycol (PEG) was of analytical reagent grade with the molecular weight of 10,000 and was used directly without further purification.

2.2. Synthesis

The procedures for the preparation of MH from the boron mud powder were as follow: the boron mud powders with the particle size of 80–150 μm were dissolved in 43% sulfuric acid solution at 80 °C for 2 h [31]. Then, the remaining residues SiO_2 powders were separated by filtration. $MgSO_4$ solution was obtained after the ions like Fe^{3+}, Al^{3+}, and Fe^{2+} in acid leaching filtrate were transformed into hydroxide precipitates, and the precipitates were separated by filtration. $MgSO_4$ solution was added in the different concentrations of $NH_3{\cdot}H_2O$ with or without 3% PEG at room temperature. The mixed liquor was stirred and aged for different time. Then, the precipitates were filtered, washed with distilled water, and dried at 105 °C to obtain MH.

2.3. Characterization

The X-ray diffraction (XRD) analysis was performed on a D/max 2500 diffractometer with nickel filtered Cu K_α radiation. Data were collected over a 2θ range of 10–70° with a step size of 0.02°. The surface morphology was investigated using a Hitachi S-4800 scanning electron microscope. Infrared spectra were recorded by KBr disks using a NICOLET750 FT-IR instrument. Thermal analysis was carried out on a Q50 V20.6 Build31 thermoanalyzer under N_2 and air at a heating rate of 10 K/min.

3. Results and discussion

3.1. Influence factors on the properties of MH

In the precipitation method, the shape and crystal size of MH are affected by concentration of $NH_3{\cdot}H_2O$, aging time, and the surfactant PEG.

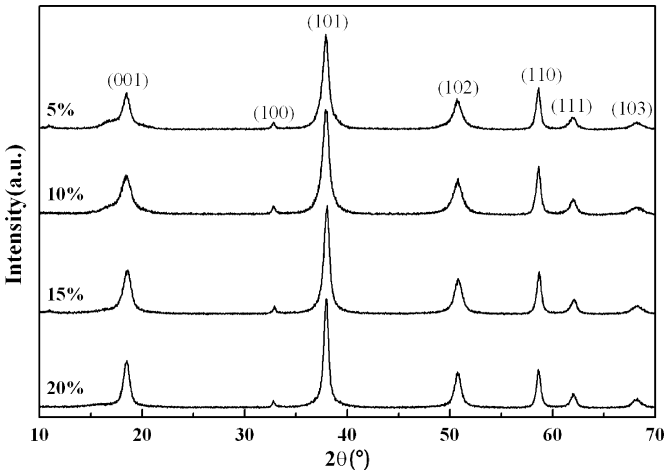

Fig. 1. XRD pattern of as-prepared MH obtained in different concentration of $NH_3{\cdot}H_2O$.

3.1.1. Concentration of $NH_3{\cdot}H_2O$

The XRD patterns of samples prepared in the different concentrations of $NH_3{\cdot}H_2O$ by aging for 30 min are shown in Fig. 1. All of the XRD patterns of samples can be indexed well for the brucite structure corresponding with ICDD 44-1482. No diffraction peaks representing other phase were detected in Fig. 1, which indicated a high purity of the brucite.

The concentration of $NH_3{\cdot}H_2O$ strongly affects the morphology of MH [23]. The SEM images are shown in Fig. 2. The flower-like MH was obtained, which existed in the form of irregular spherical aggregation. With the concentration of $NH_3{\cdot}H_2O$ increasing, the flower-like lamellas scattered into close-grained blocks. When the concentration of $NH_3{\cdot}H_2O$ solution was 20%, MH with pseudo-hexagonal blocky morphology was obtained (Fig.2d). Thus, the 5% $NH_3{\cdot}H_2O$ solution was chosen in subsequent experiments to prepare the flower-like MH.

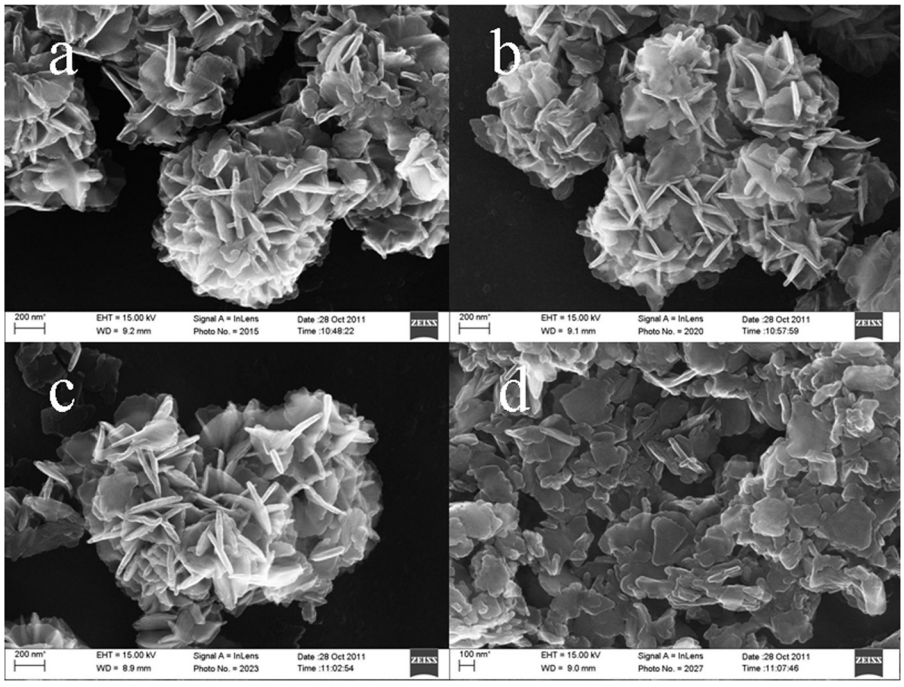

Fig. 2. SEM images of as-prepared MH obtained in different concentration of $NH_3{\cdot}H_2O$ (a) 5% $NH_3{\cdot}H_2O$, (b) 10% $NH_3{\cdot}H_2O$, (c) 15% $NH_3{\cdot}H_2O$, (d) 20% $NH_3{\cdot}H_2O$.